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Mechanical Properties of Alumina Trihydrate Filled Polypropylene/Ethylene Propylene Diene Monomer Composites for Cable Applications
(Sifat Mekanik Alumina Trihidrat Diisi Komposit Polipropilena/Monomer Etilena Propilena Diena untuk Aplikasi Kabel)

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ABSTRACT

Polymeric materials such as polypropylene (PP), polyethylene (PE) and ethylene propylene diene monomer (EPDM) are widely used as insulators for cable applications. We investigated the effect of alumina trihydrate (ATH) loading on the mechanical properties of PP/EPDM blend. Preliminary study showed that PP/EPDM (60:40) was the optimum composition. ATH filled PP/EPDM composites was prepared by using twin screw extruder. In this study, the tensile properties and hardness of the composites were evaluated. The tensile modulus and hardness increased while elongation at break and tensile strength decreased with increasing ATH content. Scanning electron microscope was used to study the morphology of ATH in PP/EPDM blend.

Keywords: Alumina trihydrate; composites; mechanical properties; PP/EPDM blend

ABSTRAK

Bahan polimer seperti polipropilena (PP), polietilena (PE) dan monomer etilena propilena diena (EPDM) digunakan secara meluas sebagai penebat untuk aplikasi kabel. Kajian ini mengkaji kesan penambahan alumina trihidrat (ATH) ke atas sifat mekanik adunan PP/EPDM. Kajian awal menunjukkan bahawa PP/EPDM (60:40) adalah komposisi yang optima. Komposit PP/EPDM terisi ATH telah disediakan dengan menggunakan penyemperit skru berkembar. Dalam kajian ini, sifat tegangan dan kekerasan komposit telah dikaji. Modulus tegangan dan kekerasan telah meningkat dengan peningkatan kandungan ATH. Sementara itu, pemanjangan pada takat patah dan kekuatan tegangan telah didapati menurun dengan peningkatan kandungan ATH. Mikroskop elektron imbasan telah digunakan untuk mengkaji sifat morfologi ATH dalam adunan PP/EPDM.

Kata kunci: Adunan PP/EPDM; alumina trihidrat; rencam; sifat mekanik

INTRODUCTION

Polymers are becoming more useful in high voltage insulations (Cousins 2000; Dorf 1993; Green 1991) due to many advantages, in comparison with the traditional insulators such as porcelain and glass (Dorf 1993; Hackam 1999; Hall 2002). The advantages include flexibility, light weight, low cost, good resistance to dielectric properties and degradation due to contamination. Besides that, polymers such as PP and PE have sufficient mechanical strength to withstand environmental and electrical stresses. Polymer blends were among one of the most interesting and useful class of materials. In recent years, elastomeric rubber-plastic blends have become technologically interesting for use as thermoplastic elastomers (TPE). TPE is a special class of polymeric material that have the properties of rubbers while maintaining the processing advantages of thermoplastics (Brydson 1995; Cousins 2000; Holden et al. 2004). TPEs are potentially recyclable and furthermore, they can be molded and extruded like plastics and therefore are faster and easier to process. Good melt flow is essential for compounding and processing the materials into wires and cables.

Among the plastics, polypropylene (PP) has a relatively higher melting temperature and therefore expected to be a good choice as an insulation material for wire and cable application. However, PP is poor in flexibility and has a low electrical breakdown strength which can limit its usefulness as insulators (Brydson 1995; Cousins 2000; Holden et al. 2004; Kurahashi et al. 2004). Therefore, PP was normally blended with elastomer to improve the flexibility. Ethylene propylene diene monomer (EPDM) is a type of synthetic elastomer with similar chain structure as that of PP and is a good choice to produce PP/EPDM blend due to its partial miscibility (Ezzati et al. 2008; Kurahashi et al. 2004; Salmah et al. 2009). By blending PP and EPDM, a TPE that combines high melting temperature and recyclability of PP with the ductility of EPDM can be produced. In addition, excellent resistance to degradation and ability to high filler loading of EPDM may reduce the product cost and make this TPE suitable for electrical applications (Allen 1989; Cheremisinoff 1998; Harper 2006; Piah et al. 2005; Strate 1986;). Many studies on the effect of blending EPDM and PP have been previously reported (Arroyo et al. 2000; Pal & Rastogi 2004; Yang et al. 2006; Zhu & Zhang 2002).

Research has been done about the brittle-ductile transition behavior of PP/EPDM blends by a percolation model and the effect of variables on the brittle-ductile transition of PP/EPDM blends was also discussed (Zheng et al. 2011).

ATH is a popular flame retardant because of its cost-benefit and low toxicity (Planes et al. 2010; Sobolev & Waycheshin 1987; Troitzsch 1990; Xanthos 2010). There are many reports on the suitability of ATH as a flame retardant for wire and cable insulation. Besides that, ATH has other advantages such as smoke suppression, optical translucency, thermal conductivity and chemical stability (Beyer 2005; Kalyon et al. 2002; Lee & Kim 2000; Olasz & Gudmundson 2005). ATH is also capable of reducing the product cost due to its relatively cheaper price than that of the polymers (Kumagai & Yoshimura 2001).

Many studies have been reported on the effect of fillers on PP/EPDM blends but limited information is known about the effect of ATH on PP/EPDM blends (Ramazani et al. 2008; Ristolainen et al. 2005; Xiao & Kibble 2008; Zhang et al. 2004). The objectives of this study were, first to determine the optimum PP/EPDM blends composition for wire and cable insulation applications based on the rheological and mechanical properties. Secondly, the objective was to investigate the effects of ATH loading on mechanical properties of PP/EPDM composites.

EXPERIMENTAL METHOD

MATERIALS PREPARATION

The materials used for preparation of ATH filled PP/EPDM composites are listed in Table 1. Test specimens were compounded by simultaneous addition of all components to Barbender Plasticoder PL 2000 counter-rotating twin-screw extruder with a barrel temperature of 185°C and a rotor speed of 20 rpm in order to determine the optimum PP/EPDM blend formulation. The extruded materials were injection molded into test specimens by using JSW (Muroran, Japan) Model NIOOB II injection-molding machine with a barrel temperature of 170-185°C. All test specimens were conditioned to condition under ambient condition for at least 48 h prior to testing. The weight ratios to determine the optimized PP/EPDM blend were: 100/0, 80/20, 70/30, 60/40 and 50/50. After a preliminary study, composite specimens were prepared with a constant ratio of the optimum PP/EPDM blend formulation at different ATH content (0, 25, 50, 75 and 100 phr). The preparation of composite specimens was carried out using the same machines and conditions of preliminary study. The blends and composites formulation used in this investigation are given in Tables 2 and 3, respectively.

TABLE 1. Material characterizations

Material	Grade	Supplier	Specification
PP	SM 240	Titan Sdn. Bhd.	Injection molding grade MFI= 25.0 g / 10 min^{-1} at 230°C
EPDM	NORDEL IP 3756P	Dow Chemical Co.	PP content: 30.5% and Mooney viscosity ML(1+4) at 125°C= 45
Silane treated ATH	HYMOD SB805SP	Akrochem Co.	$A1(OH)_3$ = 99.2% , SiO_2 = 0.005%, Median Particle size =2.6 µm, Surface area = 4-6 /gm

TABLE 2. PP/EPDM blend formulations

Sample code	P10	P8	P7	P6	P5
PP (%)	100	80	70	60	50
EPDM (%)	0	20	30	40	50

TABLE 3. ATH filled PP/EPDM composite formulations

Sample code	F0*	F25	F50	F75	F100
PP (%)	60	60	60	60	60
EPDM (%)	40	40	40	40	40
ATH (phr)	0	25	50	75	100

* F0 and P6 (Table 2) are similar in formulations

MECHANICAL TESTS

Two types of mechanical tests were carried out; tensile and hardness. Tensile test was done according to ASTM D638 using an Instron (Bucks, UK) 5567 under ambient conditions with crosshead speeds of 150 mm min^{-1} for diverse formulations of PP/EPDM blend in preliminary study and 50 mm min^{-1} for ATH filled PP/EPDM in study of ATH loading. The higher speed was chosen for PP/EPDM blend because no break occurred at the lower speed. Hardness test was carried out by using the shore type A durometer (ASKER CL 150 durometer hardness tester) according to ASTM 2240.

MELT FLOW INDEX TEST

The melt flow index (MFI) was determined according to ASTM D1238 after 10 min when a fixed pressure was applied to the molten sample via a piston and a load with total mass of 2.16 kg at a temperature of 190°C.

MORPHOLOGICAL STUDY

Scanning electron microscope (SEM), model PHILIPS XL40 was used to study the morphological surface of the blends. The microstructure of the tensile fractured samples surface was investigated in order to determine the influence of different ATH loading in PP/EPDM. The samples were sputter-coated with gold-platinum before scanning to minimize the charging effect. A bio-rad cool sputter coater, consisting of basic unit and a rotary pump was used to coat a non-conductive sample. The chamber with the samples inside was vacuumed to the pressure of below 5 Pa before the coating process started. A 20 mA sputtering current with 70 s coating time was used to obtain 6 mm deposited gold-palladium film thickness. The electron gun of SEM was energized at 25 kV and 250 times of magnification was used to capture the micrograph. The samples used in this study were pellet with approximately 25 mm in wide and 25 mm in long.

RESULTS AND DISCUSSION

PRELIMINARY STUDY

The mechanical properties of EPDM/PP blends are shown in Figures 1-3. As can be seen in Figure 1, tensile modulus and tensile strength of PP/EPDM blend decreased significantly with increasing the EPDM content. With the incorporation of 40% EPDM, the tensile modulus and tensile strength decreased to 60% and 43%, respectively, of the original values due to the dilution effect of EPDM which is less rigid on the PP matrix. Similar result was reported by Yang et al. (2006) whereby the tensile strength decreased by 47% with similar amount of EPDM content.

Figure 2 also illustrates that elongation at break increased with increasing EPDM content. The elongation at break for PP was 18% which increased to 383% for composition with 50% EPDM content in PP/EPDM blend. This is in consistent with the previous studies reported on PP/EPDM (Arroyo et al. 2000). This is expected due to high elastic behavior of EPDM. It was also reported that tensile strength decreases while the elongation at break increases with increasing EPDM content from 10% to 30% (Lu et al. 2010). Figure 3 illustrates the effect of EPDM content on the hardness of PP/EPDM blend. The figure shows a general decrease in hardness with increasing EPDM content. The hardness value of PP matrix decreased 40% with the addition of 50% EPDM. Similar trend was reported in a previous study (Pal & Rastogi 2004).

From the mechanical tests results, PP: EPDM (60:40 and 50:50) were chosen as the best choices due to the flexibility (based on the modulus), relatively good tensile strength and hardness. Furthermore, the use of EPDM should

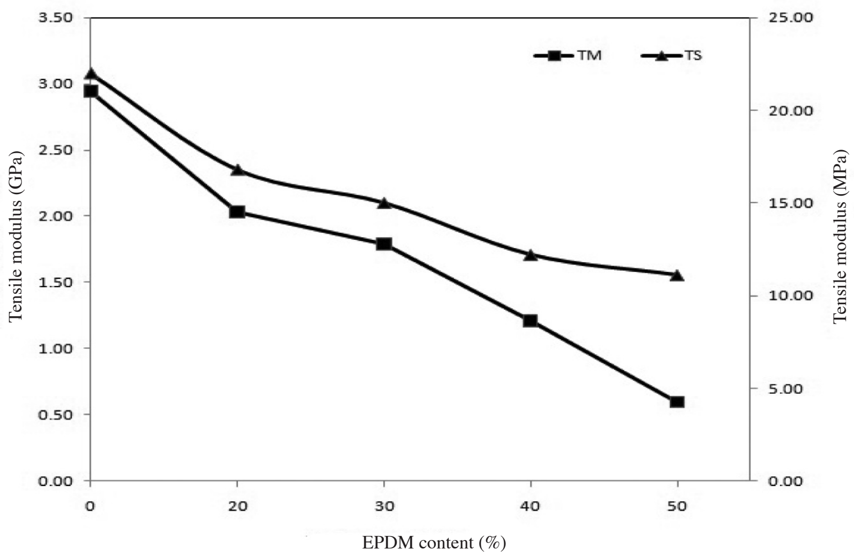

FIGURE 1. Effect of EPDM loading on tensile modulus and tensile strength of PP/EPDM blend

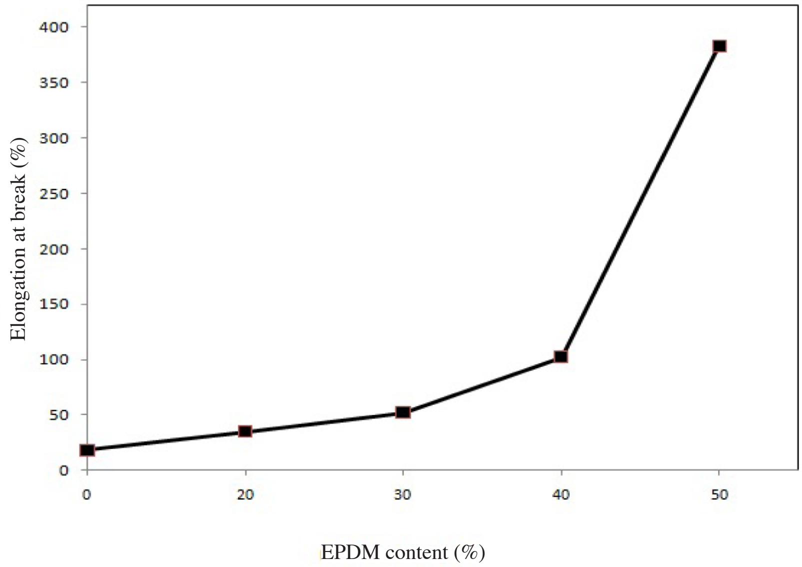

FIGURE 2. Effect of EPDM loading on elongation at break of PP/EPDM blend

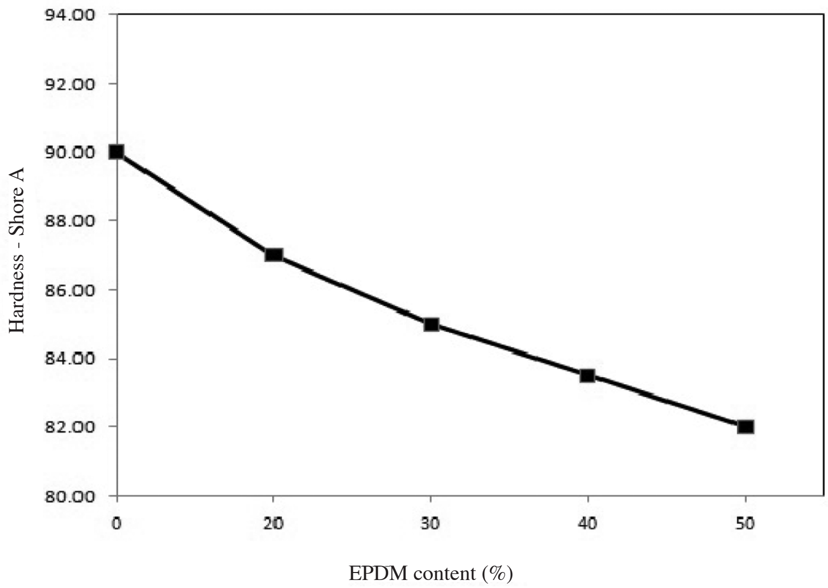

FIGURE 3. Effect of EPDM loading on hardness of PP/EPDM blend

TABLE 4. MFI of PP/EPDM blends with and without ATH

	Samples	P10	P8	P7	P6	P5
MFI	Sample/0 phr ATH	6.79	3.17	2.28	1.56	0.81
	Sample/100 phr ATH	-	-	-	0.42	0.09

be maximized as it has good insulation properties while leveraging on the good flow properties of PP. Since melt flow properties is also important for easier processing of polymeric materials into cables, MFI test was conducted in the preliminary work to determine the effect of EPDM and ATH composition on the blends and composites. Table 4 represents the MFI of PP/EPDM blends with and without ATH. As it shown, MFI of PP/EPDM blends decreases with increasing EPDM content. This result indicated that EPDM increases the melt viscosity of the blend.

The next part was to determine the effect of ATH on the selected PP/EPDM blends. The effect of 100 phr ATH on the flow properties of samples P5 and P6 was done. Table 4 illustrates that addition of 100 phr ATH to sample P5 (PP/EPDM: 50/50) decreased the MFI significantly from 0.81 to 0.09 g/10 min which resulted in poor processability of the composites. Based on this, blend of PP/EPDM (60/40) was selected as the optimum composition for the next stage of the study, i.e. to determine the effect of ATH loading on the PP/EPDM blend.

EFFECT OF ATH LOADING

MECHANICAL TESTS

The effects of ATH loading on the tensile modulus and tensile strength of ATH filled PP/EPDM composite are illustrated in Figure 4. An increase in tensile modulus is clearly observed with increasing ATH content in the PP/EPDM blend. Similar behavior was also reported by other researchers on PP and EVA matrices (Ramazani et al. 2008; Ristolainen et al. 2005; Zhang et al. 2004). The tensile modulus increased with increasing ATH content because ATH hinder the mobility of the PP/EPDM matrix. From Figure 4, it can also be seen that tensile strength decreased dramatically with the addition of 25 phr ATH to the PP/EPDM. Similar results were also reported when ATH was incorporated into PP (Ristolainen et al. 2005). The decrease is because the interaction is not strong enough for efficient transfer of stress from PP/EPDM to ATH (Ramazani et al. 2008).

As shown in Figure 5, the addition of 25 phr ATH decreased the elongation at break of PP/EPDM/ATH composite to one fourth. Also, the addition of 50 phr ATH into PP/EPDM matrix decreases the elongation at break up to 75.3% while similar results were reported by other researchers (Ristolainen et al. 2005). They found the elongation at break decreased by 93.5% with similar amount of ATH content into the PP matrix. In another study, researchers reported decrease in elongation at break for EVA/ATH when compared with unfilled EVA (Zhang et al. 2004). Xiao and Kibble (2008) also reported that the elongation at break of SEBS/PP decreased by the addition of ATH. Decrease in elongation at break in composites is due to the incompatibility between the polymer and filler. Moreover, the deformation of the filler is generally much lesser than that of the polymer matrix, thus, the filler forces the matrix to deform more than the overall deformation of the composite (Salmah et al. 2009).

Figure 6 illustrates a general increase in hardness of the ATH filled PP/EPDM composite with increasing ATH loading. Lee and Kim (2000) reported that the addition of 50 and 100 phr of ATH into the EPDM increased the hardness from 46 to 49 and 58, respectively, according to shore A hardness. Mansour et al. (2006) reported study on performance of PVC/ATH composites as insulating material and noticed that remarkable increase in the hardness of PVC composite containing 25 phr ATH.

MELT FLOW INDEX TEST

As shown in Figure 7, MFI decreased with increasing ATH loading level, an indication of viscosity increase. This means that there will be a limit on the amount of ATH loading to be added in the polymer matrix although it is expected that the higher the ATH loading the more flame retardant will be the composite (Lutz & Grossman 1988). The presence of ATH in the polymer restricts the polymer chain mobility thus increasing the viscosity. Another possible reason is the interaction between ATH (which is in treated form) with the polymer matrix. The increase in viscosity is consistent with the study reported that shows MFI was reduced from 14 to 6 g/10 min with the addition of 50 phr of ATH into the PP (Plentz et al. 2006).

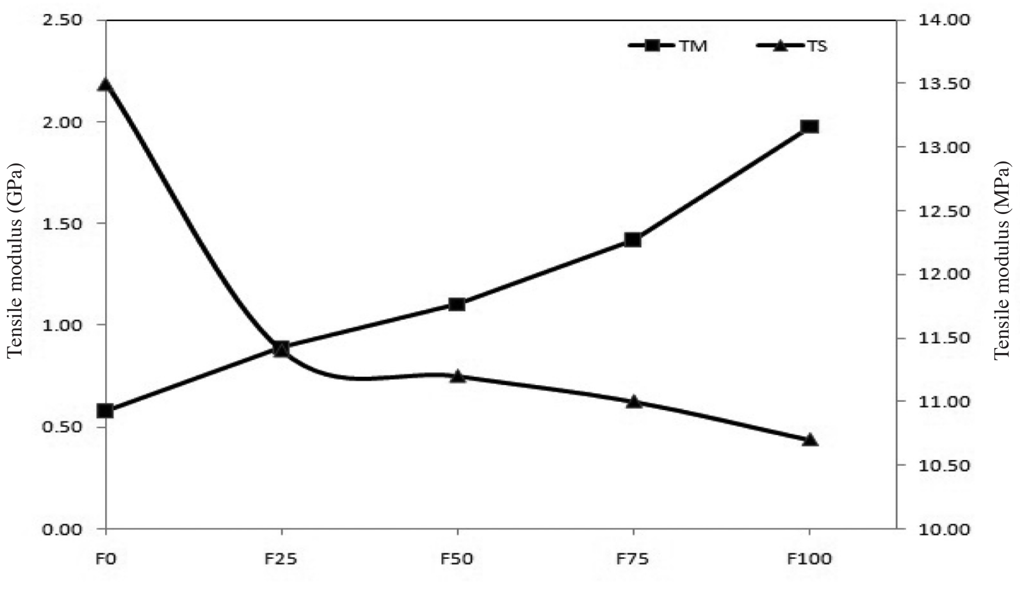

FIGURE 4. Effect of ATH loading on tensile modulus and tensile strength of PP/EPDM blend

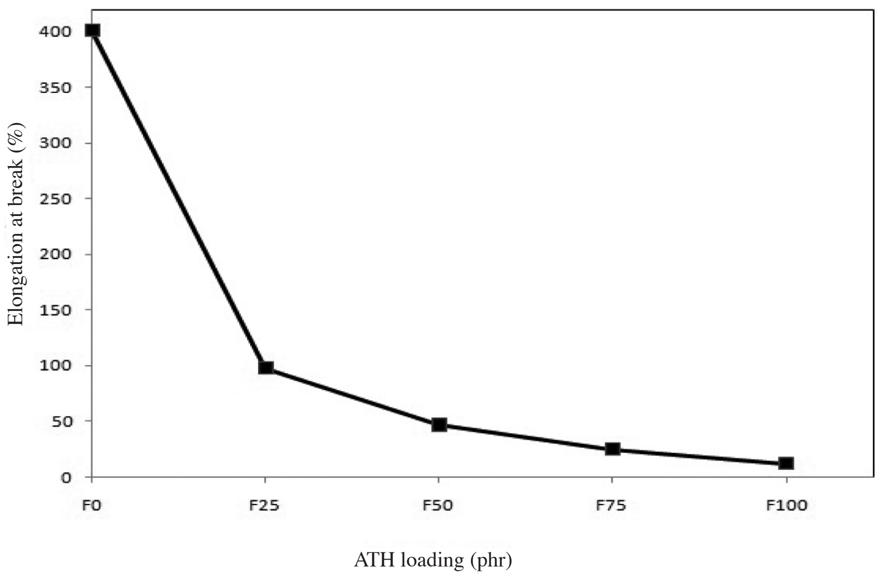

FIGURE 5. Effect of ATH loading on elongation at break of PP/EPDM blend

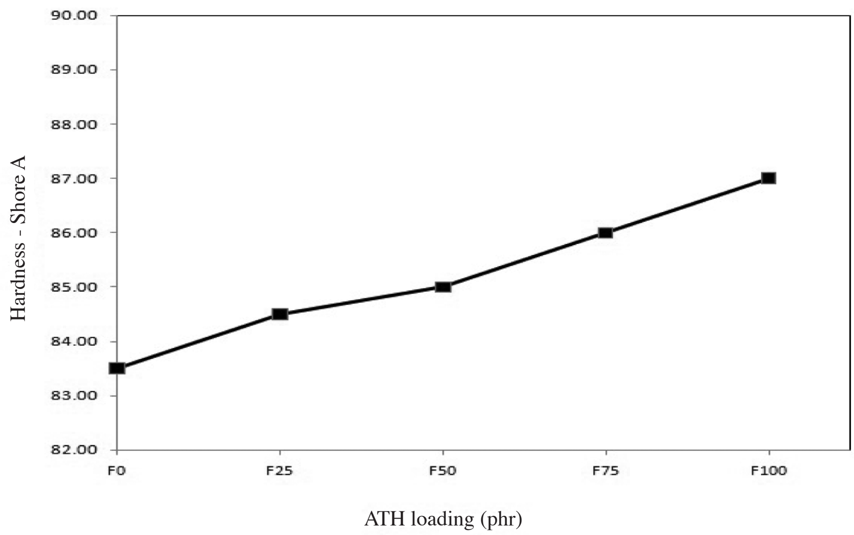

FIGURE 6. Effect of ATH loading on hardness of PP/EPDM blend

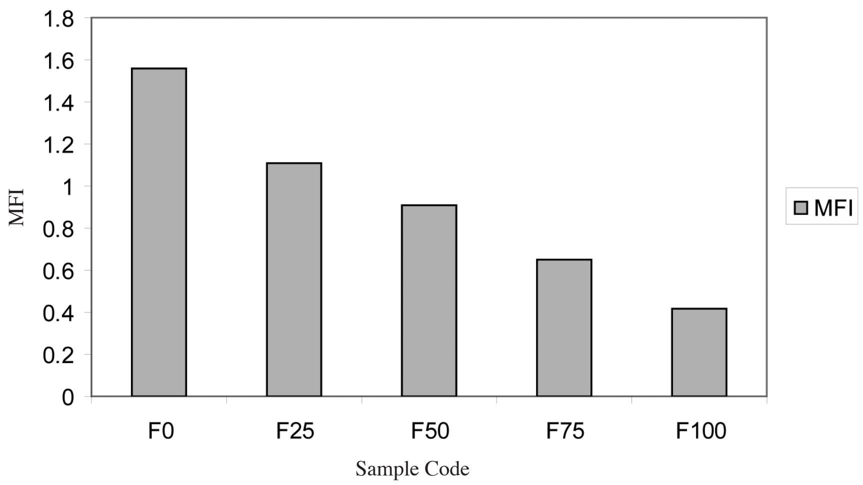

FIGURE 7. Effect of ATH loading on MFI of PP/EPDM blend

MORPHOLOGICAL STUDY

The scanning electron microscopy can be utilized to study the distribution of fillers within polymer matrix. Figure 8 illustrates the surface of PP/EPDM blend which does not contain any ATH fillers. The surfaces of PP/EPDM composites at 25, 50, 75 and 100 phr loading of ATH are shown in Figures 9-12. The ATH particles can be clearly observed as white spots within the matrix. From the micrographs it can be concluded that the ATH fillers are well distributed with the PP/EPDM matrices. It should also be noted that the tendency to agglomerate increased with increasing filler content. Adhesion quality of the filler with PP/EPDM matrix could be determined from micrograph.

CONCLUSION

The study was conducted to determine the best ATH filled PP/EPDM formulation for wire and cable application. The preliminary results showed that PP/EPDM (60:40) blend as the optimum composition based in mechanical and MFI test. The effect of ATH loading on mechanical and MFI of PP/EPDM (60:40) was then determined. The tensile modulus and hardness increased with increasing filler loading with a concomitant decrease in elongation at break and tensile strength. MFI decreased by increasing the ATH content. SEM illustrated that ATH was well distributed within the PP/EPDM matrix.

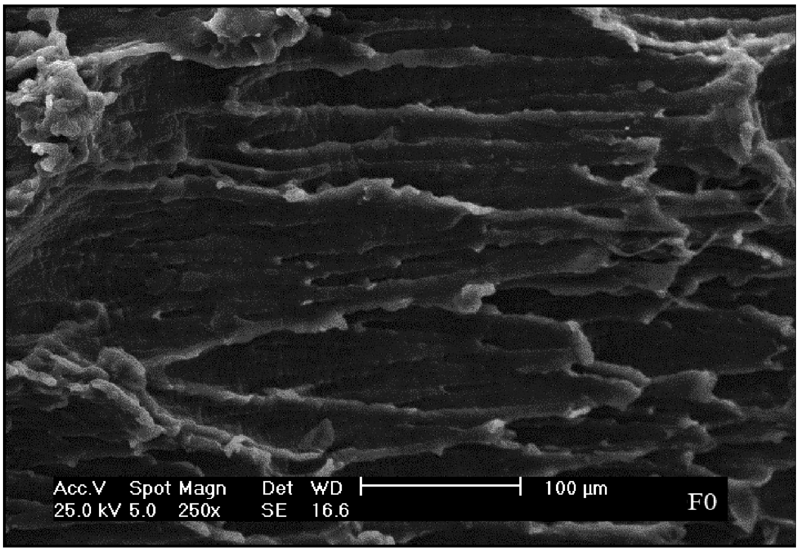

FIGURE 8. SEM micrograph of F0

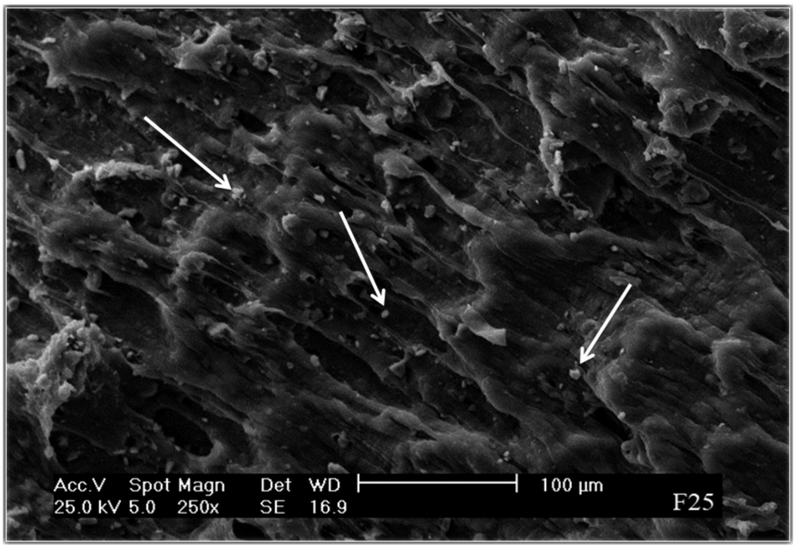

FIGURE 9. SEM micrograph of F25

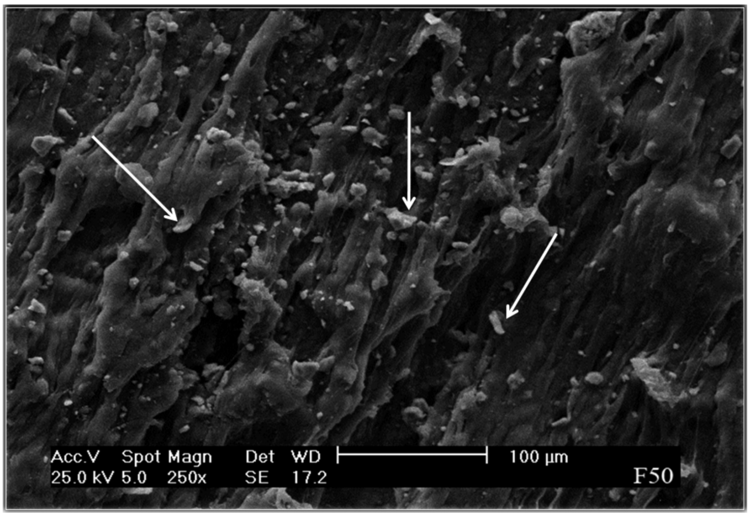

FIGURE 10. SEM micrograph of F50

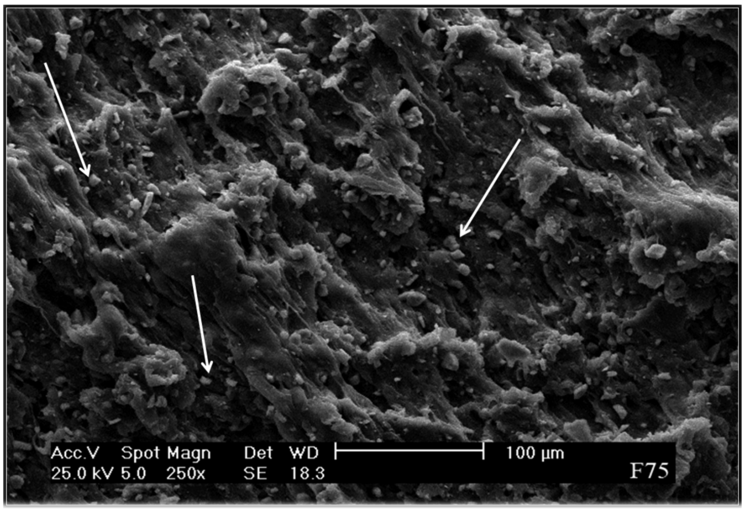

FIGURE 11. SEM micrograph of F75

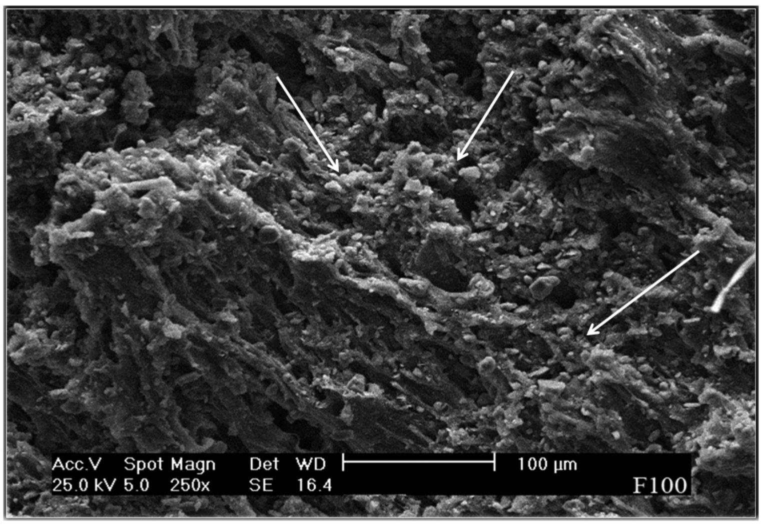

FIGURE 12. SEM micrograph of F100

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